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Indian Standard

SPECIFICATION FOR CALCIUM NITRATE FOR EXPLOSIVE INDUSTRY

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *September* 1984

Indian Standard

SPECIFICATION FOR CALCIUM NITRATE FOR EXPLOSIVE INDUSTRY

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Indian Standard

SPECIFICATION FOR CALCIUM NITRATE FOR EXPLOSIVE INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 9 March 1984, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

0.2 Calcium nitrate is used as an optional oxidizer salt in commercial blasting explosives.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for calcium nitrate for use in processing of commercial blasting explosives.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions prescribed in IS : 10081-1981† shall apply.

3. REQUIREMENTS

3.1 Description — The material shall consist essentially of calcium nitrate tetrahydrate in the form of fine colourless crystals, free from visible impurities.

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*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to commercial explosives, pyrotechnics and blasting practices.

3.2 The material shall conform to the requirements prescribed in Table 1, when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR CALCIUM NITRATE FOR EXPLOSIVE INDUSTRY

SL No. (1)	CHARACTERISTIC (2)	REQUIREMENT (3)	METHOD OF TEST (REF TO CL No. IN APPENDIX A) (4)
i)	Calcium nitrate content, percent by mass, *Min*	68·8	A-2
ii)	Iron [as $Fe(NO_3)_3$], percent by mass, *Max*	0·003	A-3
iii)	Magnesium [as $Mg(NO_3)_2$], percent by mass, *Max*	1·5	A-4
iv)	Nitrite [as $Ca(NO_2)_2$], percent by mass, *Max*	0·03	A-5
v)	Water insolubles, percent by mass, *Max*	0·03	A-6
vi)	Loss on drying, percent by mass, *Max*	30·5	A-7
vii)	*p*H (of aqueous solution)	6 to 7	A-8
viii)	Grit, percent by mass, *Max*	0·01	A-9

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in polyethylene bags and further packed in bitumenized gunny bags or HDPE woven bags. The net content of the material shall be 25 kg or 50 kg.

4.2 Marking — Each package shall be legibly and indelibly marked with the following information:

- a) Name of the material;
- b) Net mass;
- c) Manufacturer's name and/or his recognized trade-mark, if any;
- d) Year of manufacture; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

4.2.1 The package may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material from a lot, number of tests to be performed and the method of finding out the criteria of conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2, and *Table* 1)

METHODS OF TEST FOR CALCIUM NITRATE FOR EXPLOSIVE INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which effect the result of analysis.

A-2. DETERMINATION OF CALCIUM NITRATE CONTENT [$Ca(NO_3)_2$]

A-2.1 Outline of the Method — Calcium, like some other metal ions, is complexed by EDTA by choosing the proper *p*H and indicator. Using calcon as indicator, calcium can be estimated in the presence of magnesium since magnesium is precipitated quantitatively as magnesium hydroxide in the *p*H range of the reaction.

A-2.2 Reagents

A-2.2.1 *Diethylamine*

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*Specification for water for general laboratory use (*second revision*).

A-2.2.2 *Calcon Indicator* — Prepared by dissolving 0·2 g of the dyestuff in 50 ml of methyl alcohol.

A-2.2.3 *Standard EDTA (Disodium Dihydrogen Ethylenediamine Tetra Acetate Solution)* — Weigh 3·772 5 g of EDTA, dissolve in water free from polyvalent ions (distilled water for this purpose should be passed through a column of cation exchange resins in the sodium form) and make up to a volume of 1 000 ml. Concentration of the resulting solution will be 0·01 M. (If the EDTA is not of analytical reagent grade, then the solution prepared needs to be standardised against standard zinc chloride solution or magnesium chloride solution which is prepared from analytical reagent grade material or corresponding metal pellets of analytical reagent grade.)

A-2.2.4 *Sample Solution* — Weigh about one gram of calcium nitrate tetra hydrate accurately, dissolve in water free from polyvalent ions, filter and make up to 100 ml.

A-2.3 Procedure — Pipette out 10 ml of the sample solution (*see* **A-2.2.4**) into 250-ml conical flask. Add about 40 ml of water and 5 ml of diethylamine, giving the mixture a *p*H of about 12·5. Under these conditions, magnesium, if present, precipitates quantitatively as the hydroxide. Add four drops of calcon indicator to the solution and titrate with standard EDTA solution with shaking until the colour changes from pink to a pure blue.

A-2.4 Calculation

$$\text{Calcium nitrate } [\,Ca(NO_3)_2\,]\text{, percent by mass} = \frac{1{\cdot}64 \times VM}{A}$$

where

V = volume in ml of standard EDTA solution,

M = molarity of standard EDTA solution, and

A = mass in g of material taken (**A-2.2.4**).

A-3. DETERMINATION OF IRON

A-3.1 Outline of the Method — Ferric (but not ferrous) iron reacts with thiocyanate to give a series of intensely red coloured complexes which remain in true solution. In the colorimetric determination, excess of thiocyanate should be used since this increases the intensity and also stability of the colour. Strong acid (hydrochloric or nitric acid concentration 0·052 to 0·05 M) should be present to suppress the hydrolysis of ferric iron.

A-3.2 Reagents

A-3.2.1 *Potassium Thiocyanate Solution* – Dissolve 20 g of potassium thiocyanate in 100 ml of water.

A-3.2.2 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-3.2.3 *Standard Solution of Iron* (*Ferric*) — Dissolve 0·864 g of ferric ammonium sulphate in water, add 10 ml of concentrated nitric acid and dilute to one litre. 1·0 ml of this solution is equivalent to 0·1 mg of iron (Fe).

A-3.3 Procedure — Dissolve accurately 5 g of the material in 1 : 1 water-nitric acid mixture and evaporate to nearly dryness to expel excess of acid. Dilute slightly with water, oxidise any ferrous ion to the ferric state with dilute potassium permanganate solution (2g/l), adding slowly dropwise until a slight pink colour remains after stirring well. Make up the solution to 250 ml or other suitable volume. Place 50 ml of the solution in a Nessler cylinder, and 5 ml of thiocynate solution and 2 to 4 ml of 4 N nitric acid. Add the same amounts of reagents to 50 ml of water contained in a similar Nessler cylinder and run in standard iron solution from burette (use long glass rod with flattened end for mixing) until colours are matched. Note the exact volume (X ml) of the standard iron solution added. Repeat determination using 50-X ml of water. Comparison of the standard and unknown should be made soon after preparation since the colour may fade on standing for a long time.

For estimating the concentration of ferric iron more accurately, the use of photoelectric colorimeter may be resorted to. A filter showing maximum transmission at or near 480 nm should be used. Standard solutions of ferric iron which are in the range of the concentration of ferric iron likely to be present in calcium nitrate sample are prepared and the transmittance of each such solution measured with photoelectric colorimeter. A reference curve is then prepared by plotting the concentration *versus* transmittance. Now the transmittance of the sample solution is also measured and from the reference curve, the concentration of ferric iron [as $Fe(NO_3)_3$] is directly read.

A-3.4 Calculation

$$Fe(NO_3)_3 \text{ content, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of ferric iron as $Fe(NO_3)_3$ as found out from the reference curve, and

M = mass in g of the material taken for the test.

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*Specification for nitric acid (*second revision*).

A-4. DETERMINATION OF MAGNESIUM

A-4.1 Outline of the Method — Calcium is precipitated as calcium oxalate and magnesium, if any, is estimated by complexing with EDTA using eriochrome black T.

A-4.2 Reagents

A-4.2.1 *EDTA* — 0·01 M (*see* **A-2.2.3**).

A-4.2.2 *Eriochrome Black T* — Dissolve 0·2 g of the dyestuff in 15 ml of triethanolamine and 5 ml of absolute ethanol.

A-4.2.3 *Standard Magnesium Solution* — 0·01 M. Weigh 6·1 g of magnesium sulphate heptahydrate and dissolve in 50 ml of water. Make up to 250 ml with water in a volumetric flask.

A-4.2.4 *Ammonium Oxalate*

A-4.2.5 *Ammonium Chloride*

A-4.2.6 *Dilute Ammonium Hydroxide* — (1 : 1 by volume).

A-4.2.7 *Ammonia — Ammonium Chloride Buffer Solution* — *p*H 10. Add 142 ml of concentrated ammonium hydroxide to 17·5 g of ammonium chloride and dilute to 250 ml with water.

A-4.2.8 *Dilute Hydrochloric Acid* — 1 : 1 by volume.

A-4.2.9 *Calcium Precipitating Buffer Solution* — Dissolve 6 g of ammonium oxalate in about 100 ml of water, add 144 g of ammonium chloride and 13 ml of concentrated ammonium hydroxide and dilute the resulting solution to 1 litre.

A-4.3 Standardization of EDTA Solution — Pipette out 25 ml of standard magnesium solution into a conical flask and dilute to 100 ml with water. Add 5 ml of buffer followed by the addition of 6 drops of eriochrome black T indicator and titrate with EDTA solution until the colour changes from red to pure blue.

1 ml of 0·01 M EDTA = 0·243 2 mg of Mg.

A-4.3.1 *Procedure* — Weigh 1 g of calcium nitrate into a 250-ml beaker and add 40 ml of water. Add 5 ml of dilute hydrochloric acid slowly with constant stirring. Once the material is dissolved, add another 5 ml of dilute hydrochloric acid. Add a few drops of methyl red indicator. Heat the solution to boiling. Add a clear solution of 6 g of ammonium oxalate dissolved in 100 ml of water slowly with constant stirring. To the hot solution (70-80°C) add dilute ammonium hydroxide 1 : 1 dropwise until the solution becomes fairly alkaline. Digest on water bath for 1 hour.

A-4.3.2 Decant the supernatant liquid through No. 42 Whatman filter paper and collect the filtrate. Transfer the precipitate quantitatively to the filter paper and wash three times with 25-30 ml of water. Collect the washings along with the filtrate. Evaporate the solution to 100 ml. Add 2 ml of buffer solution followed by the addition of 3-4 drops of eriochrome black T indicator. Titrate the solution with EDTA solution at 40°C until the colour changes from red to pure blue. (The last traces of reddish shade should disappear at the end point. Complex formation does not take place instantaneously. Titration, therefore, should be conducted slowly near the end point.)

A-4.4 Calculation — Calculate magnesium as magnesium nitrate on the basis that one millilitre of 0·01 M. EDTA solution is equivalent to 1·096 4 of magnesium nitrate [$Mg(NO_3)_2$].

A-5. DETERMINATION OF NITRITE

A-5.0 Two methods are prescribed, namely, colorimetric method and volumetric method. Colorimetric method is recommended for routine analysis. In case of dispute, volumetric method shall be the referee method.

A-5.1 Colorimetric Method

A-5.1.1 *Outline of the Method* — When dilute solutions of sulphanilic acid and α-naphthylamine are acted upon by nitrous acid, a red colouration is produced which may be used for the colorimetric determination of nitrites. The full colour does not appear at once. A satisfactory colour comparison can be made after 10-15 minutes provided the sample and standard are treated in the same manner.

A-5.1.2 *Reagents*

A-5.1.2.1 *Sulphanilic acid reagent (solution 1)* — Dissolve 0·6 g of sulphanilic acid in 100 ml of 20 percent (V/V) dilute hydrochloric acid.

A-5.1.2.2 *α-naphthylamine reagent (solution 2)* — Dissolve 0·48 g of α-naphthylamine in 100 ml of 20 percent (V/V) dilute hydrochloric acid.

A-5.1.2.3 *Standard nitrite solution* — Dissolve 4·93 g of sodium nitrite in water and dilute with deionised water. Dilute 10 ml of standard solution to one litre with deionised water.

A-5.1.2.4 *Sodium acetate* (*2 M*) — Dissolve 16·4 g of anhydrous sodium acetate in water and dilute to 100 ml with water.

A-5.1.3 *Procedure* — Place the quantity of the sample containing about 0·03 mg or less of nitrite in a 50-ml volumetric flask. Add 1 ml of sulphanilic acid reagent, mix well and allow to stand at least for 3 minutes but not more than 10 minutes at room temperature in diffused light. Introduce 1 ml of α-naphthylamine reagent and 1 ml of 2 M sodium acetate solution to act as buffer (*p*H 2 to 2·5). Dilute to volume and mix well. Use a photoelectric colorimeter with a green filter and measure transmittance. Construct a reference curve with known nitrate standards, preferably in the nitrite nitrogen range of the sample and measure the concentration of the nitrite in the sample solution from the reference curve.

A-5.1.4 *Calculation* — The concentration of nitrite is directly read from the reference curve and calculated as $Ca(NO_2)_2$.

A-5.2 Volumetric Method

A-5.2.1 *Reagents*

A-5.2.1.1 *Potassium permanganate solution* — N/50.

A-5.2.1.2 *Sulphuric acid* — (1 : 1).

A-5.2.2 *Procedure*

A-5.2.2.1 Weigh accurately about 30 g of calcium nitrate and transfer to a 100-ml volumetric flask and dissolve with shaking in 40 ml of water. Make up the volume to the mark with water. Stopper the flask and shake well. Take up the solution in a burette.

A-5.2.2.2 Take 2·5 ml of freshly prepared N/50 potassium permanganate solution in a conical flask. Add 5 ml of 1 : 1 sulphuric acid. Dilute to 40 ml with water and heat to around 60°C and titrate against calcium nitrate solution taken in the burette till the pink colour disappears.

A-5.2.3 *Calculation*

1 ml of IN $KMNO_4$ = 0·033 03 g of calcium nitrite.

A-6. DETERMINATION OF WATER INSOLUBLES

A-6.1 Procedure — Weigh about 25 g of calcium nitrate tetrahydrate into a 250-ml beaker, dissolve in adequate quantity of water and allow to stand until the solution attains ambient temperature. Filter the solution through a preweighed sintered glass crucible (G 4), wash the insolubles on to the crucible with small quantities of water until such time the filter is free from calcium nitrate. Dry the glass crucible in an air oven at 110°C to constant mass and kept in a desiccator.

A-6.2 Calculation

$$\text{Water insolubles, percent by mass} = \frac{M}{M_1} \times 100$$

where

M = mass in g of the dried residue, and

M_1 = mass in g of the material taken for the test.

A-7. DETERMINATION OF LOSS ON DRYING

A-7.1 Procedure — Weigh about 1 g of the material accurately into a glass weighing bottle with lid closed in order to avoid absorption of water and keep it in a muffle furnace at 230°C with the lid removed. The material is kept until such time that on cooling in a desiccator constant mass is obtained. The anhydrous material is weighed after closing the sample container.

A-7.2 Calculation

$$\text{Loss on drying, percent by mass} = \frac{M - M_1}{M} \times 100$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material after drying.

A-8. DETERMINATION OF *p*H

A-8.1 Procedure — Dissolve 10·0 g of the material in 100 ml of water and measure the *p*H of the solution by means of a suitable *p*H meter using glass electrodes.

A-9. DETERMINATION OF GRIT

A-9.1 Procedure — Weigh about 50 g of the material into a 500-ml beaker, dissolve in adequate quantity of water and allow to stand until the insoluble material settles. Decant the supernatent liquor and filter the rest using a filter paper. Ignite the filter paper with the residue in a muffle furnace. Boil the residue for about 30 minutes with

20 ml aqua regia. After cooling the mixture, drain off the aqua regia and wash the residue thoroughly with water. Dry the residue in an air oven and sieve it through 125 micron IS sieve. Weigh the portion retained on the sieve and test for soda glass scratching. If it scratches soda glass then report it as percent grit.

A-9.2 Calculation

$$\text{Grit, percent by mass} = \frac{M \times 100}{M_1}$$

where

M = mass in g of the residue retained on 125-micron IS sieve, and

M_1 = mass in g of the material taken for test.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF CALCIUM NITRATE FOR EXPLOSIVE INDUSTRY

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be drawn in an exposed place.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The samples shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (*see* **3.2**) and the date of sampling.

B-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — In a single consignment of the material all the containers of the same type and size and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch, type and sizes shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE N	SAMPLE SIZE n
(1)	(2)
Up to 15	3
16 ,, 25	4
26 ,, 50	5
51 ,, 100	7
101 and above	10

B-2.1.2 In order to ensure randomness of selection, random number tables (*see* IS : 4905-1968*) shall be used. In case such tables are not available, the following procedure is recommended for use:

'Arrange the containers in the lot in a systematic order and starting from any one count them as 1, 2, 3,........, etc up to r and so on, where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every *r*th container thus counted shall be withdrawn to constitute the sample.'

---

*Methods for random sampling.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a small representative portion of the material not less than 100 g in mass.

B-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly, to form a composite sample of mass not less than 150 g. The composite sample shall be divided into 3 equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample.

B-3.3 This remaining portion of the material from each container shall be divided into 3 equal parts and transferred to separate bottles, giving full identification particulars of the samples on the bottles. The material in each bottle constitutes an individual sample. One of these three sets (each containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

B-3.4 The referee test samples consisting of a composite sample and a test of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of moisture, matter insoluble in water and total calcium nitrate shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all the remaining characteristics given in col 2 of Table 1 shall be carried out on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 From each set of the individual test results for moisture, matter insoluble in water and total strontium nitrate, the mean (X) and range (R) of test results shall be computed separately (the range being defined as the difference between the maximum and the minimum values of the test results).

B-5.1.1 The lot shall be declared as conforming to the requirements of moisture and matter insoluble in water, if ($X - 0{\cdot}6R$) as calculated from the test results is less than or equal to the relevant maximum value specified in Sl No. (i) and (ii) of Table 1.

B-5.1.2 The lot shall be declared as conforming to the requirements of total calcium nitrate if (X-0·6R) as calculated from the relevant test results is greater than or equal to 97·0.

B-5.2 For declaring the conformity of the lot to the specification requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in Table 1.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 $kg.m/s^2$
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress,	pascal	Pa	1 Pa = 1 N/m^2